



Column leaching process of rare earth and aluminum from weathered crust elution-deposited rare earth ore with ammonium salts



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Abstract: In order to better understand the leaching process of rare earth (RE) and aluminum (Al) from the weathered crust elution-deposited RE ore, the mass transfer of RE and Al in column leaching was investigated using the chromatographic plate theory. The results show that a higher initial ammonium concentration in a certain range can enhance the mass transfer process. pH of leaching agent in the range of 2 to 8 almost has no effect on the mass transfer efficiency of RE, but plays a positive role in the mass transfer efficiency of Al under strong acidic condition ($\text{pH} < 4$). There is an optimum flow rate that makes the highest mass transfer efficiency. The optimum leaching condition of RE is the leaching agent pH of 4–8, ammonium concentration of 0.4 mol/L and flow rate of 0.5 mL/min. The mass transfer efficiencies of RE and Al both follow the order: $(\text{NH}_4)_2\text{SO}_4 < \text{NH}_4\text{Cl} < \text{NH}_4\text{NO}_3$, implying the complexing ability of anion.

Key words: column leaching process; weathered crust elution-deposited rare earth ore; rare earth; aluminum; ammonium salt; mass transfer

1 Introduction

Weathered crust elution-deposited rare earth (RE) ore, also named ion-adsorbed RE ore, is a unique mineral resource in China due to the rich middle and heavy RE [1]. For the tremendous commercial value, great attention is paid to the exploitation of this ore. It is widely distributed in southern China, such as Jiangxi, Guangdong, Fujian, Hunan, Yunnan, Guangxi and Zhejiang [2]. The main mineral compositions of the weathered crust elution-deposited RE ore are quartz, potash feldspar, plagioclase, kaolinite and white mica. In a warm and humid climate, original rocks containing RE are weathered and clay minerals are formed by biological, chemical and physical effects. During this weathering process, REs in the minerals are dissociated to be hydrated or hydroxyl-hydrated RE ions and further adsorbed on clay minerals with the migration of natural water [3]. As a result of the specific occurrence state, RE can be leached out from weathered crust elution-

deposited RE ore with ammonium or sodium salt by ion-exchange method [4].

The leaching process of RE from weathered crust elution-deposited RE ore is a mass transfer process. The cations in the leaching agent diffuse from solution to the ore particles, and then the RE ions adsorbed on the ore particles are replaced and transferred into the solution [5]. TIAN et al [6–8] reported that the kinetics of RE leaching was controlled by inter-diffusion and the mass transfer efficiency would affect the leaching of RE. In the leaching process of RE, impurity ions, especially Al, accounting for 35%–75% of total amount of impurities in the leachate, are also exchanged into the leachate. The impurities in leachate would decrease the quality of RE product, increase the consumption of precipitator and even destroy the crystal forming of carbonate RE precipitation [9]. Currently, most researches focus on the leaching techniques, but limited literatures exist on the leaching mass transfer process of RE, let alone impurities [10,11]. Therefore, it is vitally necessary to investigate the mass transfer process of both

RE and impurities (Al) to reduce the consumption of leaching agent, increase the leaching efficiency of RE, and reduce the content of impurities in the leachate [12].

Due to the strong exchangeability with RE, minor environmental pollution and low cost, ammonium salts are generally recognized as the optimal leaching agents at present. Ammonium sulfate ((NH₄)₂SO₄), ammonium chloride (NH₄Cl) and ammonium nitrate (NH₄NO₃) as the common ammonium salts are widely used in the actual RE recovery [13,14]. CHI and TIAN [1] discussed the leaching efficiencies of RE and impurity using the three different ammonium salts, which indicated that the leaching efficiency both followed the order of (NH₄)₂SO₄<NH₄Cl<NH₄NO₃. It provides important theoretical basis on the choice of an advisable leaching agent from the point of leaching efficiency. It may be one-sided for the selection of leaching agents in the actual RE recovery. The comparison on the mass transfer process of RE and impurities leaching with the three ammonium salts will benefit to explore the leaching regularity, guide roundly the exploitation of RE ores, reveal the effects of anions (SO₄²⁻, Cl⁻ and NO₃⁻) and search a novel leaching agent.

In this study, the mass transfer of RE and Al in the leaching process by the three ammonium salts was investigated in the fixed bed column. The effects of leaching agent pH, ammonium (NH₄⁺) concentration and flow rate on the mass transfer efficiencies of RE and impurities (Al) were studied and results were evaluated by chromatographic plate theory. The effects of anions (SO₄²⁻, Cl⁻ and NO₃⁻) on the mass transfer of RE and Al were also discussed.

2 Experimental

2.1 Materials

The experimental RE ore sample is original RE ores collected from Dingnan (DN) County in Jiangxi Province of southern China. The RE ore sample was mixed thoroughly and dried in an oven at 50 °C for 8 h.

All chemicals in this study were purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, with analytical grade. The used deionized water was produced by an ultra-pure water system (Super series, Nison Instrument Co., Ltd., Shanghai, China).

2.2 Column leaching experiments

The experiments were processed at ambient temperature (25 °C) in a glass column with an inner diameter of 45 mm and a column length of 500 mm, which can be seen as a chromatographic column. 250 g of dried RE ore sample was packed uniformly in the glass column and the packed ore height was measured. A sand core filter plate was fixed at the bottom of the

leaching column to retain soil particles and two layers of filter paper were put on the upper of the RE ore sample to resist preferential flow. The leaching agent was added from the top of the leaching column at a constant flow rate by a peristaltic pump. Leachate was collected from the bottom of the leaching column and the concentrations of RE and Al were analyzed by titration method.

2.3 Analytical methods

The total content of RE in the leachate was determined by EDTA titration using xylenol orange as indicator and hexamethylene tetramine as buffer. After that, excess EDTA solution was added into the studied liquid sample and placed in a water bath at 90 °C for 10 min. Then, the content of Al was analyzed by back titration using a known concentration of zinc solution. The error of the titration method was ±2%.

3 Results and discussion

3.1 Characterization of RE ore

The main chemical composition of the RE ore was analyzed by X-ray fluorescence (Axios advanced, Panalytical B.V.) and the results are listed in Table 1. It reveals that the RE ore contains 0.10% RE and 19.53% Al. The occurrence states of RE and Al in RE ore were determined with the sequential fractionating extraction method [15] and shown in Tables 2 and 3. For RE, it exists as aqueous soluble state, ion-exchangeable state, colloid sediment state and mineral state. The exchangeable RE grade is 0.090%, demonstrating that 90.00% of RE in this ore exists as ion-exchangeable state. The partitioning of the exchangeable RE in the leachate was analyzed by inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7700x, Agilent Technologies Inc.) and the results are shown in Table 4.

Table 1 Main chemical compositions of RE ores (mass fraction, %)

REO	Al ₂ O ₃	MnO ₂	ZnO	CaO	MgO	K ₂ O	SiO ₂	P ₂ O ₅
0.10	19.53	0.01	0.01	0.02	0.52	3.54	58.09	0.20
SO ₃	TiO ₂	Fe ₂ O ₃	Rb ₂ O	SrO	ZrO ₂	BaO	Loss	
0.04	1.40	8.07	0.02	0.01	0.02	0.04	8.36	

Table 2 Occurrence states of RE in rare earth ore

Phase state	Mass fraction/%	Phase distribution rate/%
Aqueous soluble	8.32×10 ⁻⁶	8.32×10 ⁻³
Ion-exchangeable	0.090	90.00
Colloid sediment	0.0032	3.20
Mineral	0.0068	6.80
Full phase	0.10	100

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